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Synthesis and structure of 2′-substituted 1-(1,3-dioxan-5-yl)uracils. Positive role of the Eu(fod)3 nmr shift reagent

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Abstract

The configuration of 1-(2-R-1,3-dioxan-5-yl)uracils and the conformation of the dioxane ring in these compounds were investigated by 1H NMR spectroscopy with the aid of the Eu(fod)3 shift reagent. It is shown that the dioxane ring exists in the preferred chair conformation with an axial orientation of the pyrimidine ring; this is confirmed by the resonance of the 5′-Ha proton in the form of a broad singlet with υ1/2υ8.5 Hz. An analysis of the spectral peculiarities of the synthesized compounds made it possible to establish the orientation of the substituents attached to the second C2 steric center. The three-dimensional structure of 1-(2, 2-dimethyl-1-3-dioxan-5-yl)uracil was determined by an x-ray diffraction study, and the axial orientation of the pyrimidine ring was confirmed. It is shown that significant flattening of the carbon part of the ring (ψ = 46.6 °) is observed in this molecule. An intramolecular (C6...O1, = 3.05 Å) hydrogen bond was observed in the molecule of this compound. © 1982 Plenum Publishing Corporation.

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